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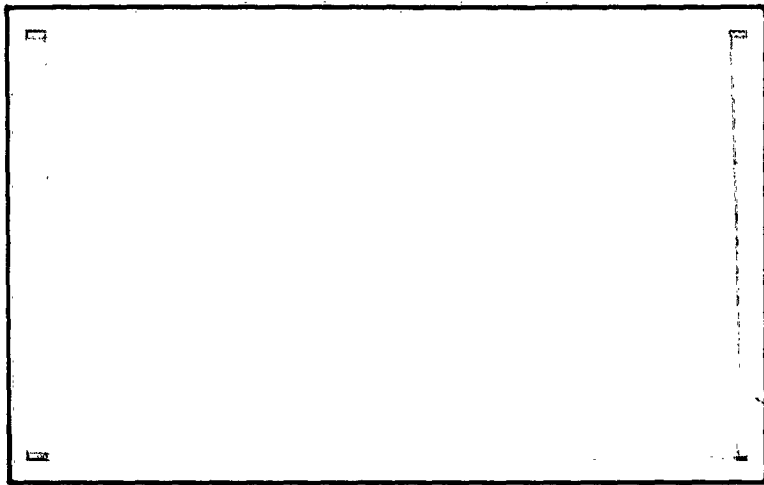
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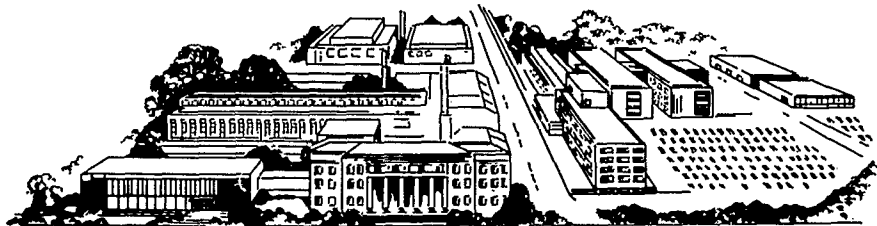
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⑥ DEVELOPMENT OF ANALYTICAL TECHNIQUES  
FOR THE DETERMINATION OF MINUTE  
QUANTITIES OF SELECTED ELEMENTS  
IN BERYLLIUM, ⑦ - ⑤ NA

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# ABSTRACT

Solid spark mass-spectrographic and emission-spectrographic methods ~~have been~~<sup>were</sup> developed for the determination of trace metallic impurities in beryllium. The analysis of trace amounts of nonmetallic impurities, especially oxygen, ~~is~~<sup>was</sup> less well established, and work is being continued on the development of analytical techniques for the analysis of minute traces of impurities in beryllium. Most emphasis is being given ~~now~~ to the less well-established analysis of the nonmetallic impurities.



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# DEVELOPMENT OF ANALYTICAL TECHNIQUES FOR THE DETERMINATION OF MINUTE QUANTITIES OF SELECTED ELEMENTS IN BERYLLIUM

by

W. M. Henry

## INTRODUCTION

Work has been continued this period on the development of analytical techniques and measurements that can provide accurate determinations of trace impurities. These include spark-source mass spectrography, emission spectrography, activation analysis, residual-resistivity measurements, conductometric carbon, Kjeldahl nitrogen and chemical sulfur, and possibly vacuum-fusion analysis.

Experimental work has shown that spark-source mass spectrography is the most sensitive and over-all rapid technique for determining all trace impurities and has the added advantage of requiring very little sample material. This latter is quite advantageous in working with research quantities of metal. The same sample material as used for mass spectrography is used for the electrical measurements which indirectly can determine total impurity contents at parts per trillion or lower concentrations. An impurity-concentrational procedure (based on separating beryllium from impurities by its evolution as the basic beryllium acetate) followed by an emission-spectrographic-solution method provides accurate detection of most metallic impurities at the low ppm level. While considerably more tedious and time consuming to use as well as requiring much larger samples than the mass-spectrographic method, this technique provides good accuracy since suitable standards can be synthesized readily. The activation analysis work, which has been limited to oxygen determination, has been found to provide good sensitivity on finely divided beryllium and effort is being extended to relate the results obtained to bulk samples which are of more direct interest. Carbon and nitrogen can be determined accurately at about the 10-ppm level and effort is being continued to increase these sensitivities as well as that of the sulfur determination. Vacuum-fusion work using a platinum-bath technique has been carried out primarily to provide a check on the accuracies of oxygen determinations obtained by the more sensitive techniques of activation and mass spectrography. However consideration is being given to use of isotopic dilution techniques to extend the vacuum-fusion sensitivity.

## PROGRAM STATUS

Two analytical methods have been developed for the analysis of trace amounts of metallic impurities in beryllium. The spark mass-spectrographic method provides fractional parts per million sensitivity. Results obtained mass spectrographically on individual elements have been intercompared with those obtained by other analytical methods and it has been found that visual reading of the mass-spectrographic plate provides comparable values at least within a factor of 2 even at elemental concentrations

in the low ppm range. Considerable attention has been given to methods of sample cleaning and mass-spectrographic operation so as to obtain equivalent data on the nonmetallic impurities. Lack of sufficient corroborative analytical data, especially on oxygen, has prevented full evaluation of the mass-spectrographic method on these elements. However useful mass-spectrographic analysis and residual-resistivity measurements have been obtained on very high-purity beryllium specimens too small in amount and too pure to be analyzed completely by other techniques.

The second technique for trace metallic impurity analysis, a chemical-concentration emission-spectrographic method developed under this program, has been used to check the validity of the mass-spectrographic results. While not providing for the analysis of as many impurity elements nor for as great a sensitivity, this technique can provide good accuracy by its utilization of a large sample and readily synthesized reference standards.

The accurate determination of low amounts of nonmetallic impurities is still under development. Work is being continued to increase the detection limits of the chemical carbon, nitrogen, and sulfur methods. Considerable effort as described below is being given to the oxygen analysis. No difficulty has been encountered in the determination of 1 ppm and lower amounts of hydrogen by vacuum extraction. However no work has been done thus far in investigating the applicability of the mass-spectrographic method for hydrogen.

### Materials

Two additional types of beryllium metals were acquired to supplement the materials presently being used in the analytical development program. These additional metals are a bulk quantity, 75 grams, of Pechiney Cast-Extruded SR Grade rod and several lots of Franklin Institute zone-refined beryllium. The Pechiney SR Grade is of an over-all purity somewhat between that of the other two beryllium materials presently being used in the program — Nuclear Metals Incorporated distilled metal and Berylco UKAEA equivalent cast metal. The amount of the Pechiney material is ample to subdivide and carry out developmental and experimental work using all of the analytical techniques being investigated under this program and interrelate the analytical data obtained.

The Franklin Institute zone-refined beryllium materials were obtained only in very small amounts — each lot consisting of two rods about 1/2 inch long and about 1/8 inch in other dimensions. This amount of material is suitable for the solids mass-spectrographic analysis, electrical-resistivity measurements and perhaps one or two additional analytical measurements such as carbon, nitrogen, oxygen or hydrogen determinations.

### Oxygen Analyses

As anticipated, the evaluation of analytical methods for the determination of oxygen in beryllium thus far has given the most difficulty. Since the main objective of this program is to develop methods for analyzing elements accurately at the 1-ppm level and lower, most of the effort on oxygen thus far has been directed toward activation analysis

and mass spectrography. A lesser effort has been made on vacuum-fusion methods primarily to aid in the evaluation of results obtained by these other methods.

The activation analysis method being investigated is one using the nuclear reaction  $^{6}\text{Li}(t, n)^{8}\text{F}$ . The tritrons in the reaction are obtained from neutron irradiation of lithium through the reaction  $^{6}\text{Li}(n, \alpha)^{3}\text{H}$ . Since about 90 per cent of the  $(t, n)$  reactions occur within 5 microns of the beryllium surface, the attaining of high sensitivity by this technique requires use of a finely divided sample. However, oxygen determinations, made on samples of crushed beryllium which had received various exposures to atmospheric oxygen and water vapor, showed that contamination of the metal surfaces occurs which can alter determined values by a factor of 5. Thus, in order to obtain oxygen values representative of the bulk metal, the beryllium must be handled in an inert atmosphere. Experimental work has shown that it is possible to determine impurity oxygen concentrations at below the 10-ppm level in a 0.5-gram sample whose mean particle diameter is 200 to 300 microns. This particle size must of course be carefully measured and be relatively uniform throughout the crushed sample in order to relate the oxygen contents found to that of the bulk sample. Additionally the particle size of the oxygen standard, ammonium nitrate, must be carefully controlled. Since the tritrons which produce radioactivation have a maximum effective range in ammonium nitrate of less than 30 microns and the mean diameter of the particle size of the ammonium nitrate can be measured, a simple geometric calculation of the volume per cent of the standard within the recoil range of the tritrons is used to obtain the reference standard values. However certain presently unexplainable deviations have been encountered in the reference standard calculations when applied to larger particle sizes of the ammonium nitrate. Experimental data given in Table 1 are representative of the problems due to both atmospheric contamination and to variations in the particle size of the reference standard.

TABLE 1. OXYGEN DATA OBTAINED ON BERYLCO  
UKAEA EQUIVALENT SAMPLE

Analysis	Oxygen Found, ppm	Exposure to Oxygen-Contaminated Atmosphere Before Analysis
1	200 $\pm$ 20	Minimum
2	270 $\pm$ 70	1 day
3	440	2-3 days
4	1020(a) 3180(b)	5-6 days

(a) Based on 360- $\mu$ -particle-diameter standard.

(b) Based on 1840- $\mu$ -particle standard.

To aid in the evaluation of the effectiveness of the beryllium handling procedure, several beryllium materials were prepared in the manner used for the activation method — i. e., crushing to a small particle size in an inert atmosphere. These crushed portions were then subdivided for subsequent oxygen analysis by several analytical techniques. In addition, bulk sample analyses on the same materials are being obtained also by these several techniques. The beryllium materials being evaluated for oxygen in this work are (1) Pechiney SR Grade cast extruded rod, (2) Nuclear Metals Incorporated distilled metal and (3) Berylco UKAEA equivalent grade cast metal. The analytical techniques being used for the fine-particle and/or bulk-size beryllium samples are given in Table 2.

TABLE 2. ANALYTICAL METHODS BEING USED FOR  
OXYGEN IN BERYLLIUM ANALYSES

Analytical Method	Sample Form Used	
	Fine Particles	Bulk Sample
Activation $O^{16}$ (t, n) $F^{18}$ reaction	Yes	No
Activation $O^{16}$ (n, p) $N^{16}$ reaction	Yes	Yes
Activation machine source	No	Yes
Inert-gas fusion	Yes	Yes
Vacuum fusion	Yes	Yes
Solids mass spectrograph	No	Yes

Surface oxygen contamination is also a problem in using the mass spectrographic method of analysis. To date the procedure being used is to obtain a "clean" surface by either acid etching, rinsing and rapidly drying the sample specimens or to cleave pieces to provide fresh surfaces. In either case two small rods representing the sample are placed as rapidly as possible into the source section of the mass spectrograph which is then evacuated to a pressure of about  $1 \times 10^{-7}$  Torr and baked at about 250 C overnight. The source section is then cooled and evacuated to a pressure of about  $2 \times 10^{-8}$  Torr and the high-voltage ionization spark is played over the ends of the specimen rods for several hours prior to obtaining the mass-spectrographic-sample spectra. Linearity studies made on beryllium materials assumed to be homogeneous, have shown that reproducible and uniform concentrations of oxygen can be obtained as evidenced by isotopic line densities.

Experimental work was carried out in an effort to accelerate the spark cleaning process by the use of an argon ion-bombardment technique. For this, a small flow of argon was introduced into the source section of the mass spectrograph during the spark cleaning process. The argon introduction and source evacuation conditions were varied to provide a range of pressures from  $1 \times 10^{-6}$  to  $1 \times 10^{-3}$  Torr during the experimental work. A considerable argon glow was observed around the sample electrodes and, under certain conditions, throughout the entire source chamber. However subsequent analyses of samples indicated little if any increase in the ability to remove sample surface contaminants by this type of argon bombardment.

Plans for future work on oxygen determination will depend to a certain extent on the outcome of the results obtained using the techniques described in Table 2. However the use of enriched oxygen in certain of these methods should prove useful. The use of an isotopic dilution technique in conjunction with the vacuum-fusion method looks promising especially to evaluate the extent, if any, of the regettinger action of the beryllium vapor on the released gases. Also if equilibration conditions can be attained, even on very small samples, in the vacuum-fusion method the sensitivity could be extended considerably. If the isotopic dilution technique were pursued for oxygen by the use of vacuum fusion, the advantages and disadvantages of concurrent nitrogen and carbon analyses would be examined. A prepared enriched oxygen atmosphere would be useful also in evaluating the effectiveness of the mass-spectrographic high-voltage spark in removing surface contamination prior to analysis.

### Sulfur Determination

Previous work showed a considerable discrepancy between the mass-spectrographic and the chemical results obtained on sulfur in the Berylco material. The mass-spectrographic analysis gave a value of 10 ppm, while a chemical sulfate method and a combustion method provided values of about 500 ppm as the sulfur content. Sulfur has not been detected chemically in any of the other beryllium materials being used in this program. However the present detection limit of the chemical combustion method is 100 ppm.

To investigate the possibility that the variation might be due to a very low ionization of sulfur by the mass-spectrographic high-voltage spark, a mass-spectrographic analysis was carried out on a standard steel sample using the same ionization parameters as used for beryllium. The mass-spectrographic sulfur results of 50 ppm checked almost exactly with the chemical analysis. Additional mass-spectrographic analyses of the Berylco sample have given somewhat higher but variable results, indicating a possibility of lack of homogeneity, especially of sulfur.

Two sets of determinations for sulfur in the Pechiney Cast Rod and the Berylco (UKAEA) beryllium metal samples were made. In the first set the method of C. L. Luke [*Anal. Chem.*, **21**, 1369 (1949)] was used directly. While the blank was low (0.6 and 1.5 micrograms of sulfur) the results on the samples were low and inconsistent.

The second set of determinations were made by separating the sulfate from the beryllium on an alumina column, eluting the sulfate with  $\text{NH}_4\text{OH}$ , converting to  $\text{H}_2\text{SO}_4$  by passing through an ion exchange column, and finally applying Luke's method of reduction and measurement. The results are shown below.

<u>Sample</u>	<u>Sulfur Found (ppm)</u>
Pechiney Cast Rod	61
	56
	17
Berylco (UKAEA)	47
	29
	46

(Blanks = 11.0 and 13.5 micrograms)

The results are still somewhat inconsistent, but do indicate a sulfur content of about 50 parts per million. This confuses the picture as to the true sulfur content of the Berylco metal as well as the methods used to analyze it, since previously about 500 ppm had been found in the material by combustion and by a titration method.

A series of experiments were made in an attempt to lower the detection limit of sulfur in beryllium. The sulfur was separated by means of an alumina column as reported by Robert Lim (Report Number UCRL-6639). Measurement of the sulfur was made by the method of Luke.

In the initial experiment, solutions containing known amounts of sulfate were carried through the procedure to check recovery and the size of the blank in this low range. The results are given in Table 3.

TABLE 3. SULFUR RECOVERY FROM PURE SOLUTIONS

Micrograms Sulfur Added	Micrograms Sulfur Found	Micrograms Sulfur Net
3.3	33.3	6.8
10.6	46.0	19.5
19.8	52.5	26.0
33.0	60.5	34.0
39.6	71.0	44.5
Blank	26.5	--

Our experience with Luke's method applied directly to metals has indicated a normal blank to be about 1 to 2 micrograms. Since the blank obtained with the proposed method is 10 to 20 times higher we felt that sulfate must be introduced in some of the reagents. To check this theory a series of experiments were planned and executed.

To establish a base for comparison of blank values, a standard solution and blank were prepared with redistilled HCl. These were evaporated, transferred to flasks, and evaporated to a low volume. The reduction, evolution, and measurement steps were then followed. Results are shown in Table 4.

TABLE 4. RESULTS OF DIRECT REDUCTION AND EVOLUTION

Micrograms Sulfur Added	Micrograms Sulfur Found	Micrograms Sulfur Net
19.8	33.5	22.5
Blank	11.0	--

Ammonium hydroxide is used to elute sulfate from the alumina column and is a possible source of sulfate. To check this, the usual amount of  $\text{NH}_4\text{OH}$  was added to two standard solutions of sulfate and the ion-exchange separation was made. These solutions were then evaporated and treated as before. A blank was carried through the procedure at the same time. Results are shown in Table 5.

TABLE 5. EFFECT OF  $\text{NH}_4\text{OH}$  ON BLANK AND SULFATE RECOVERY

Micrograms Sulfur Added	Micrograms Sulfur Found	Micrograms Sulfur Net
19.8	39.2	24.8
19.8	36.0	21.6
Blank	14.4	

Since the blank obtained in this experiment is essentially that of the base value, it appears that  $\text{NH}_4\text{OH}$  does not contribute substantially to the blank.

Perchloric acid was considered as one possible source of sulfate. Since an alumina column is used to adsorb sulfate from solution, it seemed reasonable to purify perchloric acid by passing a dilute solution through an alumina column. This purified acid was then used in the procedure with the results listed in Table 6.

TABLE 6. RECOVERY OF SULFUR USING  
PURIFIED PERCHLORIC ACID

Micrograms Sulfur Added	Micrograms Sulfur Found	Micrograms Sulfur Net
19.8	71.7	44.7
19.8	63.0	36.0
Blank	27.0	--

The blank remained essentially the same as that obtained in the initial experiment, but the net recovery of sulfur was so high that some doubt was cast on the whole experiment.

Work was stopped at this point without obtaining a thorough understanding of the source of high blanks. One generalization is that manipulations and transfers increase the amount of sulfate pickup. This may be due to contamination from the air or from the columns or beakers.

At this time, the combination Lim-Luke method is not satisfactory for the determination of low-level (less than 0.01 per cent) amounts of sulfur in beryllium metal. A better understanding of the factors involved in the relatively high blank are needed before the technique can be satisfactorily applied to this problem.

#### Hydrogen Analysis

Three beryllium materials were analyzed for hydrogen content using a 20-minute extraction at 1050 C in a copper bath. The beryllium content of the copper bath was held below 12 weight per cent. The results obtained as given in Table 7.

TABLE 7. HYDROGEN ANALYSIS OF BERYLLIUM SAMPLES

Sample	Sample Weight, g	H <sub>2</sub> Found, ppm
Nuclear Metals Inc. distilled metal	1.016	3.3
Pechiney Cast extruded rod	1.144	4.2
Berylco vacuum- cast metal	0.941	8.3



Electrical-Resistivity Measurements

Electrical-resistivity measurements have been made on thirteen beryllium materials ranging from the fairly impure grade of Berylco UKAEA equivalent metal through distilled and zone-refined materials. The approximate total impurity range of these materials, as determined mass spectrographically, is from 1500 to 150 ppm atomic. The residual-resistivity ratios obtained coincide closely with the total impurity contents in these materials. This data is being examined presently to see if definite relationships can be established in respect to any single or groups of impurity elements. However the results obtained thus far indicate that residual-resistivity measurements provide a very rapid and simple method of determining the over-all purity of a beryllium metal.

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